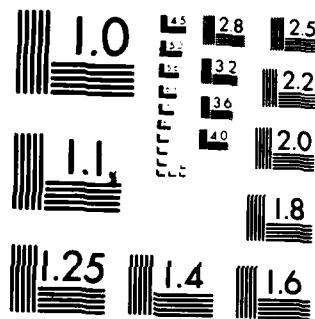


AD-A132 896 PRE-EXISTING POLARIZATION AND INFLUENCE OF ELECTRODE 1/1
MATERIALS ON PVF2 EL. (U) NATIONAL BUREAU OF STANDARDS
WASHINGTON DC POLYMER SCIENCE AN. A S DEREGGI ET AL.
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TECHNICAL REPORT NO. 20

Pre-existing Polarization and Influence of Electrode

Materials on PVF₂ Electrets as Determined by

Thermal Pulse and Pyroelectric Methods

by

Aime S. DeReggi, Frederick I. Mopsik, Martin G. Broadhurst,

and Sidney B. Lang

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**PRE-EXISTING POLARIZATION AND INFLUENCE OF ELECTRODE MATERIALS
ON PVF₂ ELECTRETS AS DETERMINED BY THERMAL
PULSE AND PYROELECTRIC METHODS**

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ABSTRACT

A number of polyvinylidene fluoride (PVF_2^{II}) electrets were prepared with different permutations of gold and aluminum electrodes and poled with DC fields up to 160 MV/m^{-1} at room temperature. Polarization distributions were measured by the thermal pulse method and pyroelectric coefficients were determined. Quantitative measurements were made of a significant level of polarization in nominally unpoled PVF_2^{II} and a contact electrification mechanism was proposed. No consistent effects of electrode materials on polarization distribution were found. PVF_2^{II} poled at room temperature has its highest polarization near the center of the thickness in contrast to the results on samples poled at elevated temperatures and cooled inhomogeneously with the field applied.

INTRODUCTION

The unique pyroelectric and piezoelectric properties of oriented and poled polymers such as polyvinylidene fluoride (PVF₂) have been studied by large numbers of workers during the past 15 years (see reviews by Broadhurst and Davis¹, Das-Gupta², Marcus³, Lovinger⁴, and Kepler and Anderson⁵). In order to produce significant pyroelectric and piezoelectric activity, uniaxially- or biaxially-stretched polymers must be poled. The process generally consists of deposition of electrodes on both sides of the film, and the imposition of high ($>100 \text{ MV m}^{-1}$) electric fields at room- or elevated temperatures followed by cooling either with or without an applied field⁶⁻¹⁰. Corona¹¹ and plasma poling¹² techniques have also been used.

Many researchers have studied the mechanism of poling and the influence of various poling parameters by analyzing pyroelectric and piezoelectric responses, thermally stimulated currents, IR spectra, and x-ray diffraction scans. These analyses all have the limitation that they yield results which are averages over the thickness of the polymer film. Some attempts have been made to determine polarization as a function of depth in the film thickness. Phelan *et al.*¹³ poled sandwich structures consisting of four layers of PVF or PVF₂ and measured the pyroelectric coefficient in each layer. More recently, Marcus^{14,15} constructed 8-layer sandwiches and measured the piezoelectric coefficient, achieving higher resolution. Eisenmenger and Haardt¹⁶ have used an acoustic stepwave probing technique, which yielded even higher resolution. One of the most promising techniques is the thermal pulse method suggested by Collins¹⁷⁻²⁰. The experiment consists of applying a thermal pulse by means of light flash on one surface of the material and measuring the electrical response generated by the sample as the heat diffuses across the thickness. Collins' original analysis used an analogue simulator to determine the polarization distribution. DeReggi *et al.*²¹ made the analysis much more quantitative by showing that the electrical response could be analyzed in terms of the Fourier

coefficients of the charge or polarization distribution across the sample. The method has been refined and numerically implemented by Mopsik and DeReggi^{22,23} so that it is now possible to obtain routinely the first seven to ten Fourier coefficients of the polarization distribution, enabling resolution of the order of 2.5 μm in a 25 μm -thick film.

This paper describes an application of an experimental facility for thermal pulse studies based on the numerical analysis method of Mopsik and DeReggi²³. The initial objective of this study was to examine the influence of different electrode materials on the polarization distribution since it was previously reported that the current during poling and the pyroelectric activity of PVF₂ depend on the electrode metal. In the course of the present work, it became clear that extraneous effects related to preexisting polarization in nominally unpoled samples were responsible for observed variations of the polarization distributions. Furthermore, the distributions of the polarization after poling were symptomatic of material inhomogeneity across the thickness.

EXPERIMENTAL METHOD

The experimental material studied was 25.4- μm thick biaxially-stretched PVF₂. Sheets cut from the roll were carefully marked to denote the originally convex and concave surfaces. Either aluminum or gold electrodes, 12.7 mm in diameter and 100 nm in thickness were deposited on each side of the film. The samples were mounted under slight radial tension between two close-fitting brass rings. Samples were poled in air with a DC power supply at voltages of 1, 2, 3, or 4 kV for 10 minutes at room temperature. The electrodes were covered with a thin layer of silicone oil during poling to prevent arcing. A charge amplifier was used to measure charge flow during poling. Correction of these data for capacitive charge storage and conduction permitted the calculation of polarization charge. After poling the samples were stored in a short-circuited condition overnight before further testing.

Thermal pulse measurements were made by exposing each side of the sample to a number of 80- μ s duration light pulses from a photographic flash unit. The charge flowing in the external circuit was measured by a charge amplifier and recorded with a high-speed transient recorder. The transient recorder permitted the averaging of the results of 16 to 64 light flashes. The data were manually smoothed and appropriately scaled with the transient recorder, and the information was then transferred to a cassette tape. A minicomputer processed the data and computed the first seven or eight Fourier coefficients of the polarization distribution. An x-y recorder was used to graph the spatial distribution. The polarization distribution measurements were made on a number of samples prior to poling also.

After the thermal pulse measurements were made, pyroelectric coefficients were measured using a method developed by Broadhurst et al²⁴. The samples were held with a foam pad in good thermal contact with a heavy-walled copper cell. Water at a temperature slightly below ambient was circulated through channels cut in the cell base. The pyroelectric current produced by the sample as it cooled was measured. Then warm water was circulated and the process was repeated while the sample was warmed. The pyroelectric coefficient was calculated from the reversible pyroelectric current, the rate of change of temperature, and the electrode area.

RESULTS AND DISCUSSION

Table I shows the pyroelectric coefficients of several of the samples studied early in the work. The four samples (270, 274, 279 and 282) subjected to the weak poling field of $\pm 40 \text{ MV m}^{-1}$ show the apparently anomalous feature of invariance of the sign of the pyroelectric coefficient with reversal of the poling field direction. The sign of the pyroelectric coefficient is well behaved for samples 285 and 286 poled with stronger poling fields. The anomalous results of samples 270, 274, 279 and 282 are explained by significant polarization

existing prior to poling of magnitude greater than the polarization induced by the weak poling field. A calculation assuming additivity of the preexisting and the poling-induced polarization gives a response due to preexisting polarization of the order of $0.1 \mu\text{C m}^{-2} \text{K}^{-1}$ which is comparable to the response measured in samples 285 and 286 before poling. The magnitude of the preexisting polarization varied among samples. However, the sign of the preexisting polarization was such that the originally convex surface of the film as defined by its curvature before it was taken from the roll always became charged negatively when $T > 0$.

Thermal pulse measurements revealed a polarization distribution with greater polarization near the center than on either side of the samples as shown in Figure 1. These results are consistent with a recent study with only Al electrodes²⁵. Although a field of 80 MV m^{-1} or greater reversed the prepolarization the polarization distribution was nonuniform at low fields, and fields of 160 MV m^{-1} or higher were required to give essentially uniform polarization. Intermediate fields always yielded a polarization distribution with a maximum near the center. The polarization near the originally convex surface ($X = 0$) was always less than that near the originally concave surface ($X = 1$) so that the slope of the polarization curve on the left-hand side of the diagrams was steeper than on the right-hand sides. This "signature" could even be distinguished in samples poled with fields of 160 MV m^{-1} , and it proved to be an unerring indicator of the originally convex and concave surfaces of the film.

Nakamura and Wada²⁶ have reported levels of piezoelectricity activity in undrawn and unpoled, and drawn and unpoled PVF₂ of 1 and 10% relative to that of drawn and well-poled material. They also described pyroelectric activity in unpoled material, but since the activity was observable only after the material was heated above 130 °C, its origin may not have been pyroelectric. Wang²⁷ found that drawn, unpoled films were not piezoelectric, but further rolling and annealing treatments yielded piezoelectric coefficients of between 1 and 5% of

that of well-poled material.

The origin of the pre-existing polarization is unknown. However, the possibility that it was produced by contact electrification during processing can be considered. Davies²⁸ made measurements of the surface charge produced by contact between a metallic surface and a polymer sheet. We may assume that, during processing, the unelectroded PVF₂ film came into contact with a metal roller possibly while still at elevated temperature. The difference between the work functions of the metal and the polymer could have caused a transfer of electrons which diffused into or out of a thin layer adjacent to the surface of the polymer. This space charge could have induced polarization in the film. Despite the fact that charge released at a metal-polymer interface would be expected to be neutralized in time, switching times for poling have been shown by Furukawa and Johnson²⁹ to be short enough that substantial poling could have occurred before the charges disappeared. Davies²⁸ showed that, if the injected charge is uniformly distributed to a depth λ (cm), the surface charge density σ (C cm⁻²) is given by
$$\sigma = 1.77 \times 10^{-13} \epsilon \Delta\phi/\lambda$$
 where ϵ is the dielectric constant of the polymer at and $\Delta\phi$ is the difference in work functions between the metal and the polymer (eV). The work function of PVF₂ does not seem to have been measured. However, Davies cites work functions for various polymers ranging from a low of 4.08 eV for Nylon 66 to a high of 4.85 eV for polyvinyl chloride. Assuming $\Delta\phi = 0.5$ eV and $\lambda = 0.2$ μm , a surface charge density of $\sigma \sim 0.05 \mu\text{C cm}^{-2}$ may be estimated. Broadhurst *et al.*³⁰ have shown that the pyroelectric coefficient is proportional to the polarization. The data on pyroelectric coefficient versus polarization shown in Fig. 2 is in good agreement with this model. Extrapolating Fig. 2 to a polarization of $0.5 \times 10^3 \mu\text{C m}^{-2}$ ($0.05 \mu\text{C cm}^{-2}$), a pyroelectric coefficient of $0.2 \mu\text{C m}^{-2} \text{ K}^{-1}$ would be predicted. Thus the magnitude of the preexisting polarization is consistent with that predicted by a contact electrification model. However,

the absence of information about manufacturing details do not allow ruling out other causes of prepolarization. For example, exposure to a corona discharge to control static electricity and dust attraction could also give rise to prepolarization.

Preliminary experiments were conducted to determine the effects of different electrode metals on the polarization distribution and the pyroelectric coefficients of PVF₂ electrets. The metals selected were aluminum and gold, with work functions of 4.08 and 4.82 eV, respectively³¹. A number of samples having all permutations of electrode materials and poled with fields of 80 or 160 MV m⁻¹ were prepared. The measured pyroelectric coefficients are graphed versus the polarization charge in Fig. 2. Polarization distribution curves are shown for a selection of samples in Fig. 3. In each case, position X - 0 corresponds to the surface which was the convex one on the original roll of polymer. The distributions for the 80 MV m⁻¹ poling field all have their maxima close to the center, and all exhibit the "signature" of the original polarization. No obvious effect of electrode materials appears in these data.

Sussner and Yoon³² and Takahashi et al.³³ reported an influence of electrode materials on the pyroelectricity. In both cases, samples with one gold and one aluminum electrode were studied. If the gold electrode was positive during poling, the pyroelectric coefficient was between 10 and 50% higher than if the polarity were reversed. However, the scatter in the data was very large.

Das-Gupta et al.^{34A} observed steady-state charging currents in PVF₂ samples having either gold or aluminum electrodes. No electrode effect was found. They suggested that an oxide layer might have been present between their polymer samples and the metallic electrodes, and the oxide-polymer interface may have dominated the charging current behavior.

Any electrode effect is most probably dependent upon the differences in electrode and polymer work functions. On the basis of the data of Davies²⁸,

the work function of PVF₂ is probably comparable to those of gold or aluminum.

Work functions depend very strongly upon the mechanical and chemical state of the surface, defects, and impurities. Thus, it is quite possible that the large data scatter^{32,33} and the influence or lack of influence of electrode materials may be due to large variations and even sign changes in work function differences. In order to resolve this question, extreme care is necessary to insure that all materials and techniques are completely reproducible.

In every sample tested, the polarization was higher in the central parts of the thickness than it was near either surface. Any skewness or differences between the polarization levels at the surfaces were always artifacts remaining from the prepoling state. These results are for PVF₂ poled at room temperature. The temperature of the process which caused the preexisting polarization is not known. However, Phelan *et al.*¹³ and Sussner and Dransfeld³⁵ observed that PVF₂ samples poled at elevated temperatures and cooled with the field applied had the maximum polarization adjacent to the poling anode. Marcus^{14,15} found similar results, but, in addition, noted that the polarization was a maximum near the center if the sample was cooled without an applied field. He also observed that samples poled in a temperature gradient were most strongly poled near the cooler side, regardless if it were the cathode or the anode. Other polymers gave different results. Lang *et al.*³⁶ showed the polyvinyl fluoride poled at room temperature had its maximum polarization near the cathode, and Phelan *et al.*¹³ observed a similar behavior for high-temperature poling with cooling in the presence of the field. Broadhurst *et al.*³⁷ examined a copolymer consisting of 73 wt % vinylidene fluoride and 27 wt % tetrafluorethylene. Its polarization was a maximum near the anode for room-temperature and high-temperature poling (with field applied during cooling).

The present work shows that electrode effects were not controlling ones in the development of the polarization distribution in the samples studied and furthermore were smaller than effects associated with sample inhomogeneities.

None of our results show effects attributable to conduction. Such effects are not ruled out in other materials, or at higher temperatures.

ACKNOWLEDGMENT

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Table I. Pyroelectric coefficients of unpoled or weakly poled samples.

Sample	Electrodes and polarity		Poling field	Pyroelectric coefficient ^a ($\mu\text{C m}^{-2} \text{K}^{-1}$)
	Originally Convex	Originally Concave	(MV m ⁻¹)	
270	Al(+) - PVF ₂ - Al(-)		+40	-0.082
274	Au(-) - PVF ₂ - Au(+)		-40	-0.209
279	Al(+) - PVF ₂ - Au(-)		+40	-0.095
282	Au(-) - PVF ₂ - Al(+) -		-40	-0.239
285 ^b	Al(+) - PVF ₂ - Al(-)		0	-0.115
285 ^c	Al(+) - PVF ₂ - Al(-)		+160	+23.490
286 ^b	Al(+) - PVF ₂ - Al(-)		0	-0.201
286 ^c	Al(+) - PVF ₂ - Al(-)		+80	+1.938

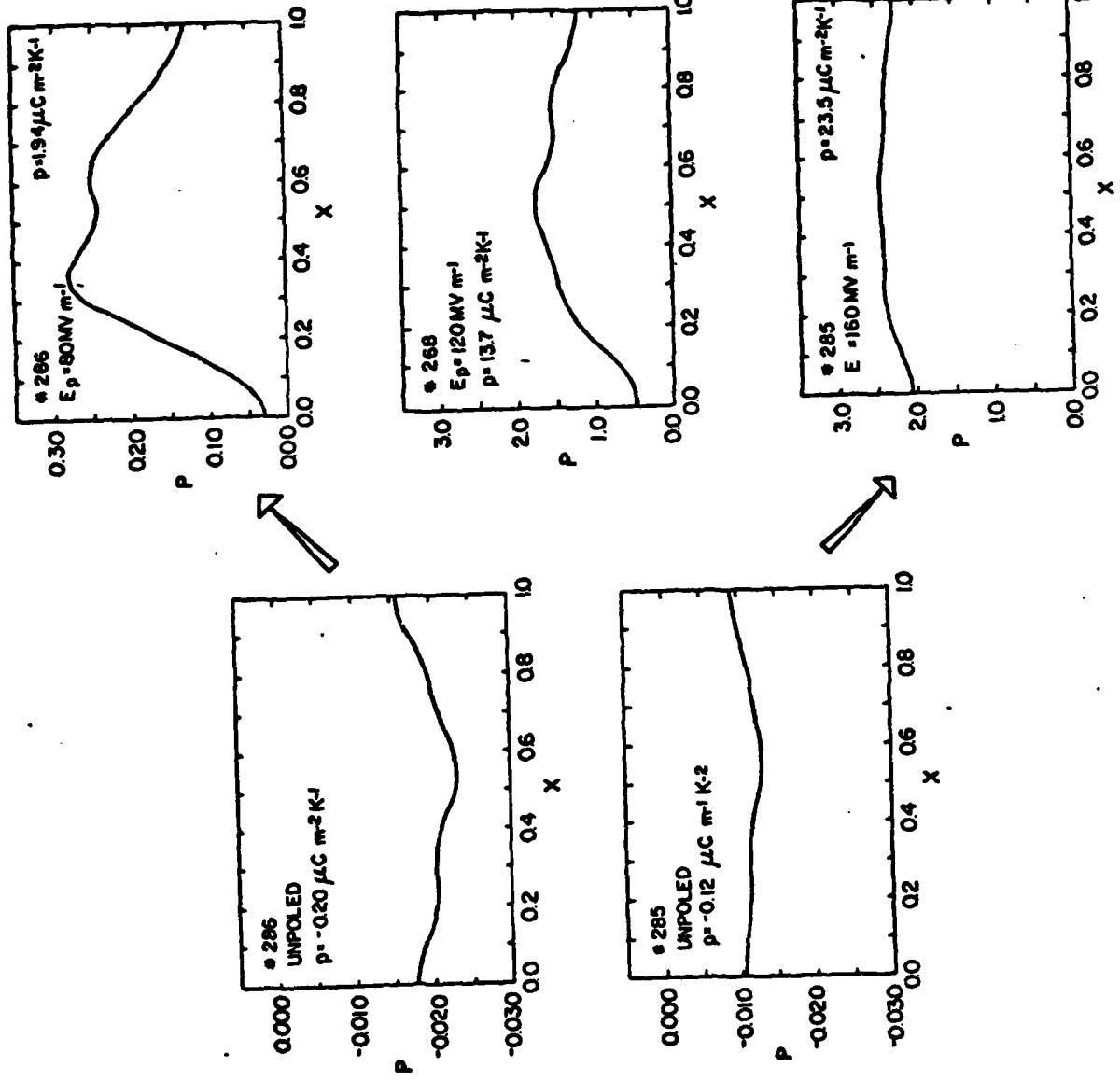
^aThe sign of the pyroelectric coefficient is the same as that of the charge induced on the originally convex side when $T > 0$.

^bPre-poling.

^cPost-poling.

FIGURE CAPTIONS

1. **Polarization distributions of unpoled (left-hand side) and poled (right-hand side) PVF_2 samples.** The surfaces which were convex on the original roll of polymer correspond to $X = 0$. Both electrodes were Al and poling was at room temperature. The arrows denote the pre- and post-poling distributions for Samples 285 and 286. The ordinates in the graphs have arbitrary units; however, the magnitudes are self-consistent among the graphs.
2. **Pyroelectric coefficient as a function of polarization for PVF_2 samples with various electrode materials.** The legend designates the anodes and cathodes during poling.
3. **Polarization distributions for various electrode materials and polarities.** The top row shows results for poling fields of 80 MV m^{-1} ; the bottom row for 160 MV m^{-1} . The surfaces which were convex on the original roll of polymer correspond to $X = 0$. The ordinates in the graphs have arbitrary units; however, the magnitudes are self-consistent among the graphs.



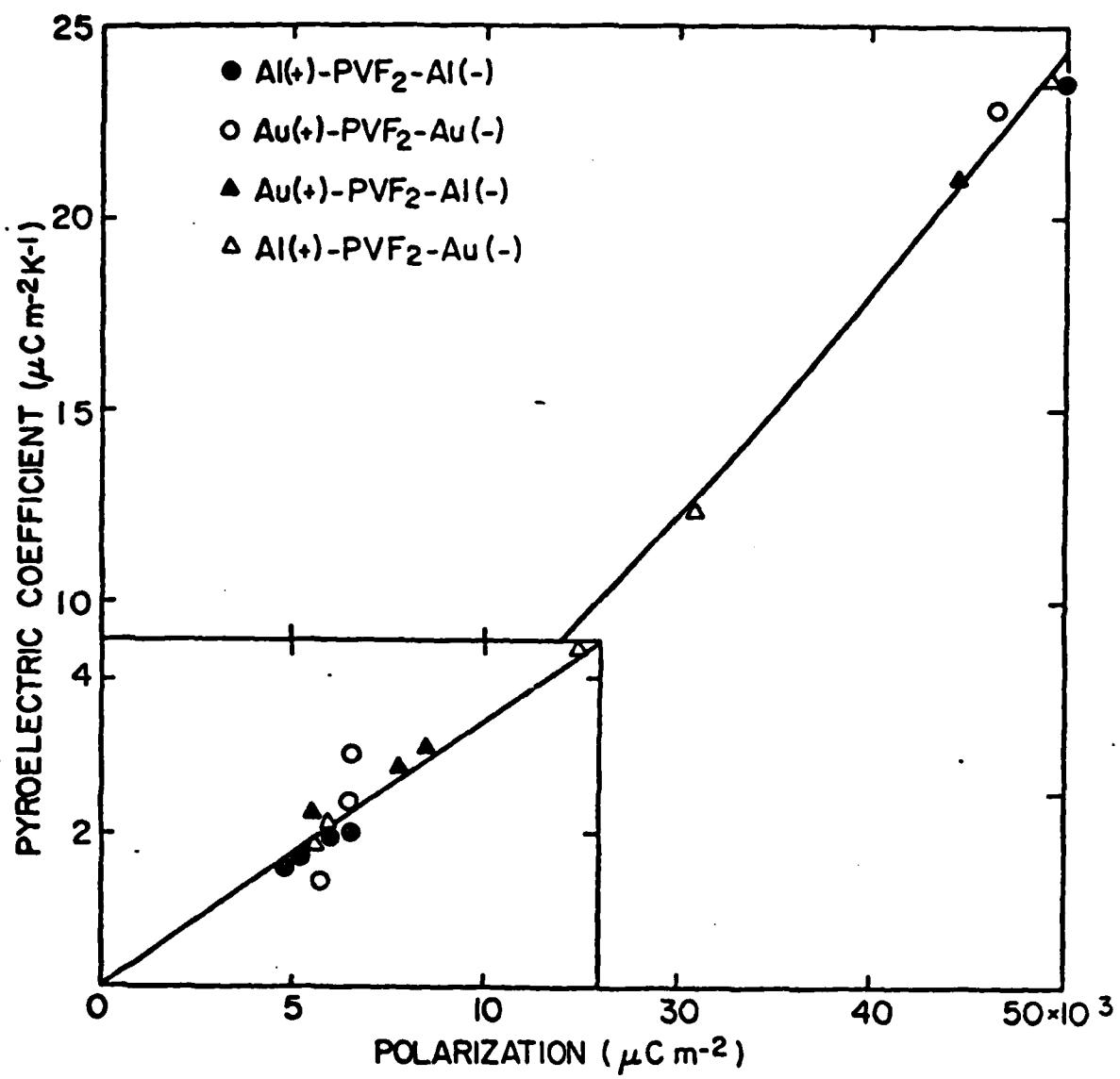
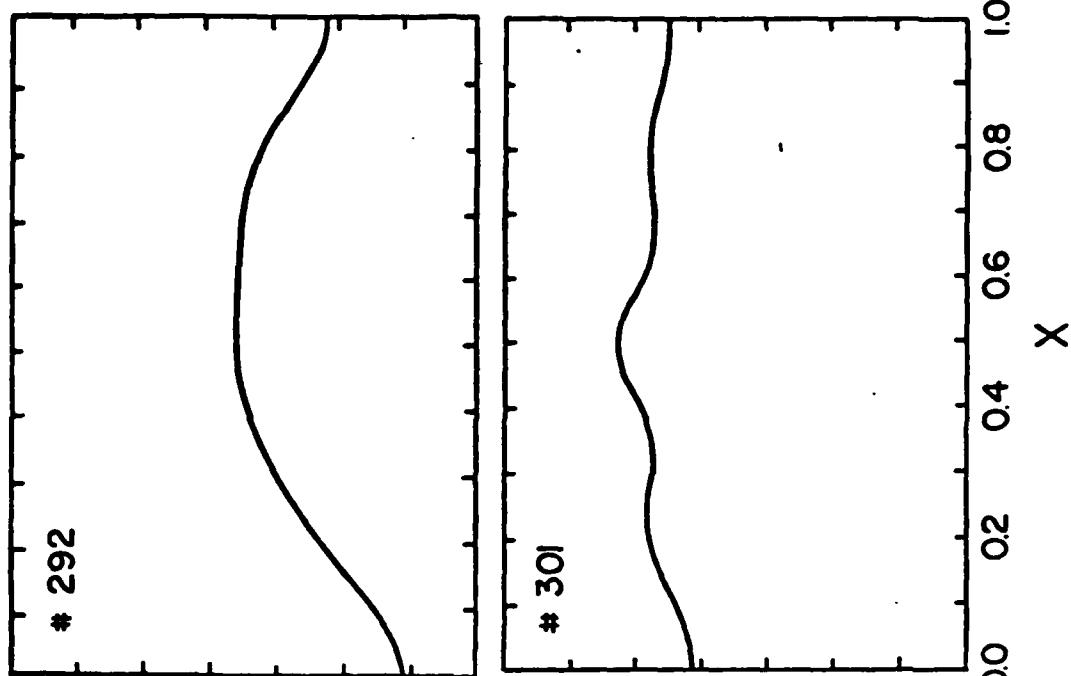


Fig. 2

Au(+) - PVF₂ - Au(-)



Al(+) - PVF₂ - Al(-)

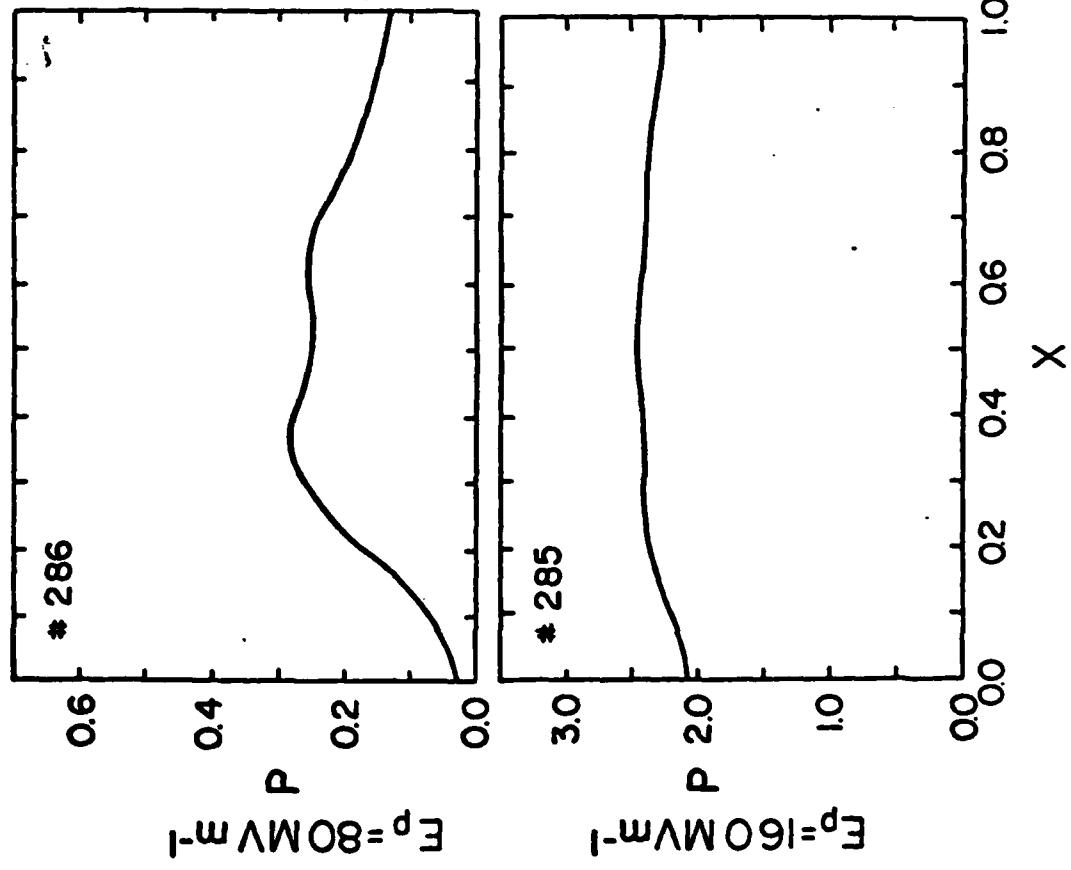


Fig. 34

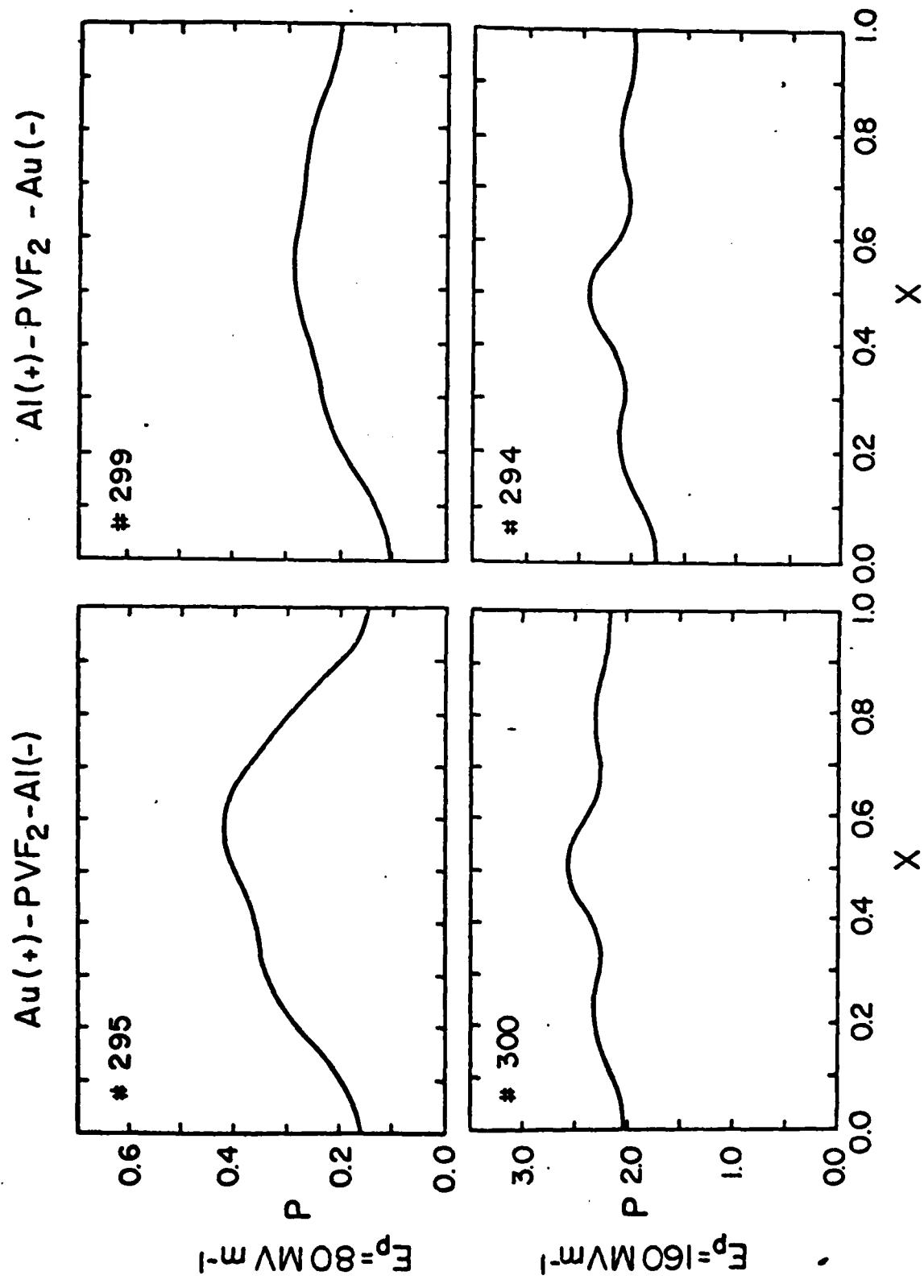


Fig. 3 b

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